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Indian Standard

METHODS OF SAMPLING OF CHEMICALS AND CHEMICAL PRODUCTS

PART II SAMPLING EQUIPMENT

Section 2 For Liquid

UDC 543·053 : 661-404

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Indian Standard

METHODS OF SAMPLING OF CHEMICALS AND CHEMICAL PRODUCTS

PART II SAMPLING EQUIPMENT

Section 2 For Liquid

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Indian Standard

METHODS OF SAMPLING OF CHEMICALS AND CHEMICAL PRODUCTS

PART II SAMPLING EQUIPMENT

Section 2 For Liquid

0. FOREWORD

0.1 This Indian Standard (Part II/Section 2) was adopted by the Indian Standards Institution on 5 May 1978, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 It may be emphasized that the most careful work in the laboratory or quantitative results may be rendered useless if care is not taken when drawing the sample. The sample which has to be truly representative of the lot, should not include material other than that to be sampled and should not change in composition before testing.

0.3 Equipment commonly used for sampling of chemicals and chemical products in the form of liquids is covered in this section. This section does not cover statistical aspects of sampling like scale of sampling, number of tests, criteria for conformity or preparation of test samples. The general requirements and precautions in sampling of chemicals and chemical products have been covered in Part I of this standard.

0.4 In the preparation of this standard, considerable assistance has been derived from BS 5309 : Part 3 : 1976 ' Methods for sampling chemical products : Part 3 Sampling of liquids ', issued by the British Standards Institution.

1. SCOPE

1.1 This standard (Part II/Section 2) deals with common sampling equipment for chemicals and chemical products in the form of liquids. It also describes the procedures to be followed in using those equipment.

2. GENERAL PRECAUTIONS AND REQUIREMENTS

2.1 It is not possible to give general directions regarding sampling equipment explicit enough to cover all cases. The guidelines given in this standard should be supplemented by judgement, skill and sampling experience. It should be ensured that the samples drawn should represent the general character and average condition of the material.

2.2 The type of sampling equipment should be chosen depending upon:

a) Type of sample (liquid);

b) Size and type of container (drum, tank, pipeline, etc);

c) Amount of sample required for tests;

d) Whether sample is in homogeneous or heterogeneous form;

e) Reactivity of sample with material of construction of sampling equipment;

f) Whether a spot sample or continuous or intermittent sample (from a pipeline) is required;

g) Whether surface or middle or bottom sample is required; and

h) Whether sampling is to be done hot, cold or at room temperature.

2.3 The whole sampling apparatus, including equipment, cord, connecting tubing and sample containers should be free from any contaminating substance. The sample container should be tightly closed immediately after sampling.

2.4 During sampling operations, the material being sampled should be protected as far as possible from adverse wind and weather. As far as possible samples should not be taken in an exposed place.

2.5 The operator engaged in sampling should have clean hands. In certain cases it may be essential for the operator to wear clean gloves to safeguard against health or other hazards. In special cases the operator may also use goggles or gas mask. Liquefied gases cause cold burns and thus should be prevented from coming into contact with skin.

2.6 Whenever possible the sampling apparatus and the sample container should be rinsed at least once with the liquid to be sampled.

2.7 The sample should be drawn through main openings or dip hatches of tanks and other bulk containers, so that direct and unconfined access to the bulk of liquid is possible. Samples should not be drawn from dip pipes or other fittings nor should gauge glasses or drain fittings be used for sampling purposes except when specified.

2.8 When the sample of a volatile product is to be transferred from the sampling apparatus into the sample container, the equipment should be up-ended into the opening of the sample container and should remain in this position until the contents have been transferred so that no unsaturated air is entrained in the transfer of the sample. The transfer of such products from the sampling apparatus to the sample container should be done immediately to avoid evaporation.

2.9 To avoid mixing up of samples collected at a time, the sample containers should be labelled and clearly marked before transferring the sample. It is preferable to use separate sampling apparatus for separate samples. In marking the container, name of sample, number, date of sampling, supplier's name, batch number and other relevant details should be mentioned.

2.10 All metal components of sampling equipment, used in a flammable atmosphere should be constructed of non-ferrous metal (to avoid sparks).

2.11 No sample container should be completely filled with liquid and sufficient ullage should always be left to allow for expansion. The ullage percent may be 20 for liquids.

2.12 Discharge of liquefied petroleum gas can give rise to static electricity and it is essential to connect the metallic bombs/cylinders to 'earth' during discharge.

2.13 Samples of material which may be affected by light or heat should be stored in a cool and dark place. The sample containers of volatile materials should be stored upside down to prevent loss of low boiling constituents; periodical examination should be made for leakage. Sample containers should be chilled prior to handling.

2.14 When samples are required from various levels in a tank or bulk container, the order of sampling should be from top downwards so that each sample is obtained before the liquid at that level is disturbed.

2.15 Sampling equipment, connecting tubing and sample container should be constructed of a material unreactive to the material to be sampled. Various materials of construction can be:

a) glass (heat resistant/borosilicate);

b) steel, stainless steel (04 Cr 17 Ni 12Mo 2) or alloy steel/plain steel, cast iron/wrought iron;

c) non-ferrous (brass, copper, lead, aluminium);

d) nylon, polyvinyl chloride (PVC), acrylonitrile butadiene styrene (ABS), polyethylene; or

e) rubber (neoprene, nitrile, synthetic).

2.16 For sampling of mobile homogeneous liquids the container selected for sampling should be mixed thoroughly by shaking or stirring or both, by suitable mixers/stirrers or by rolling (for small containers).

2.17 After closing the sample container with a tight ground glass stopper or cork, the mouth of the container may be sealed with sealing wax.

2.18 When sampling flammable liquids, there should be no naked flame or source of ignition nearby.

2.19 The sizes, dimensions and materials of construction of sampling equipment can be modified judiciously, where normal equipment is inappropriate.

3. SAMPLING EQUIPMENT FOR LIQUIDS

3.1 General — The liquid materials can be categorized as:

a) mobile liquids;
b) liquids and solids, which can be rendered mobile;
c) viscous liquids;
d) multiphase liquids;
e) liquefied gases (liquids volatile at room temperature); and
f) hazardous liquids (liquids that require special handling).

3.2 Selection — The selection of sampling equipment depends on the viscosity, heterogeneity and storage temperature of the liquid. Besides, the choice of equipment will also depend on size of sample container.

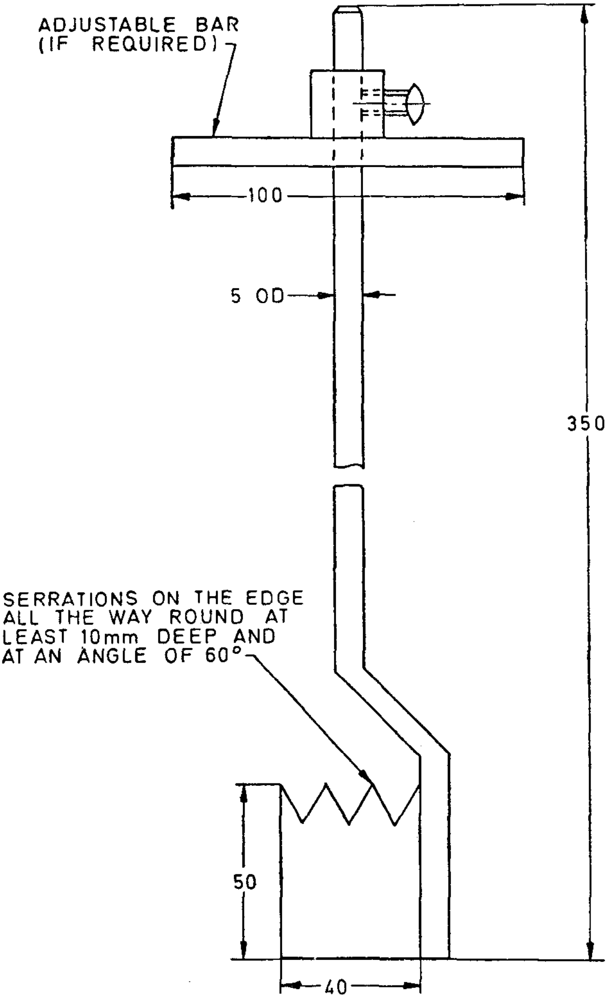
3.3 Sampling Equipment for Mobile Liquids

3.3.1 *Sampling from Small Containers* — From a small container, for example, drum, can, barrel, tin or a cask, the sample can be drawn by using a ladle, open-sampling tube, bottom-valved sampling tube or thief. The sampling equipment should be of glass, metal or a suitable plastic material (for example, polypropylene) considering the reactivity of the liquid to be sampled with the material of construction of the sampling equipment.

3.3.1.1 *Ladle*

a) *Design and construction* — A typical ladle is shown in Fig. 1. The diameter of the ladle is such that it will pass through a bunghole. The serrations on the edge should be all the way round, at least 10 mm deep and at an angle of 60°.

b) *Procedure for using the ladle* — Mix the contents of the drum preferably in closed position. Place the drum on its side with the bunghole uppermost or if the drum has an end bung stand it upright. Clean the surface around the bung and remove the bung carefully. Place the bung or cover-cap beside the bunghole with the innerside uppermost. Lower the ladle into the drum until the upper points of the serrated edge are just above the surface of the liquid. Remove the ladle before it fills completely and allow any liquid adhering to the ladle to drain off. Transfer the liquid to the marked sample container (of a material resistant to the liquid). Stopper the sample container immediately to avoid exposure of the liquid to atmosphere (lest it should absorb moisture, dust or react with oxygen, carbon dioxide, etc, present in atmosphere). Close the hole of the drum immediately after drawing the sample.

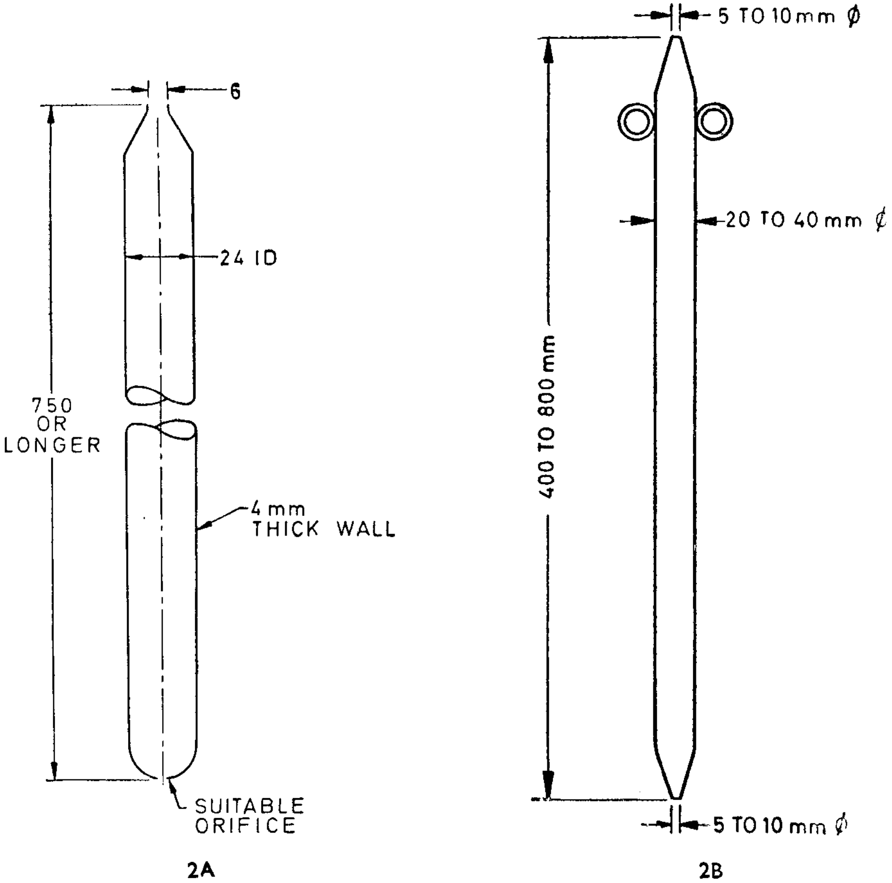

ADJUSTABLE BAR (IF REQUIRED)
100
5 OD
350
SERRATIONS ON THE EDGE ALL THE WAY ROUND AT LEAST 10mm DEEP AND AT AN ANGLE OF 60°
50
40

All dimensions in millimetres.

FIG. 1 LADLE

3.3.1.2 *Open sampling tube*

a) *Design and construction* — A few open sampling tubes made of thick glass are shown in Fig. 2A and 2B. A length of 75 cm is suitable for most drums but a longer length may be required for other containers. One end is reduced to 6 mm internal diameter for case of closure by thumb. The other end is drawn to an orifice diameter that is appropriate to the viscosity of the material to be sampled. Three orifice diameters can be conveniently used, for example, 1·5, 3 and 5 mm. The 3-mm orifice is suitable for most



All dimensions approximate in millimetres.

FIG. 2 OPEN SAMPLING TUBE

liquids having a viscosity like that of acetone, water, etc. The 5-mm orifice is convenient for liquids having a viscosity greater than that of water and less than that of glycerol. Fig. 2B shows two-rings, soldered to the opposite sides of the tube at the upper end as it makes convenient for holding the tube. The capacity of the tube can be 500 ml to 1 litre.

b) *Procedure for using open sampling tube* — Place the drum or barrel on its side with the bung up. If the drum does not have a bung, place it upright. Remove the bung and place it beside the bung hole with the lower side up. Close the upper end of the clean dry sampling tube with the thumb and lower the tube slowly to the desired depth. Remove the thumb and allow the liquid to flow into the tube. Again close the upper end with the thumb and withdraw the tube. If required the tube can be rinsed with the liquid by holding it nearly horizontal and turning it so that the liquid comes into contact with the inside walls of the tube throughout its length. Collect the sample again preferably from a depth as near to the bottom as possible. Withdraw the tube quickly and transfer the contents to the sample container marked for the purpose. Do not allow hands to come in contact with any part of the sample. Close the sample container tightly.

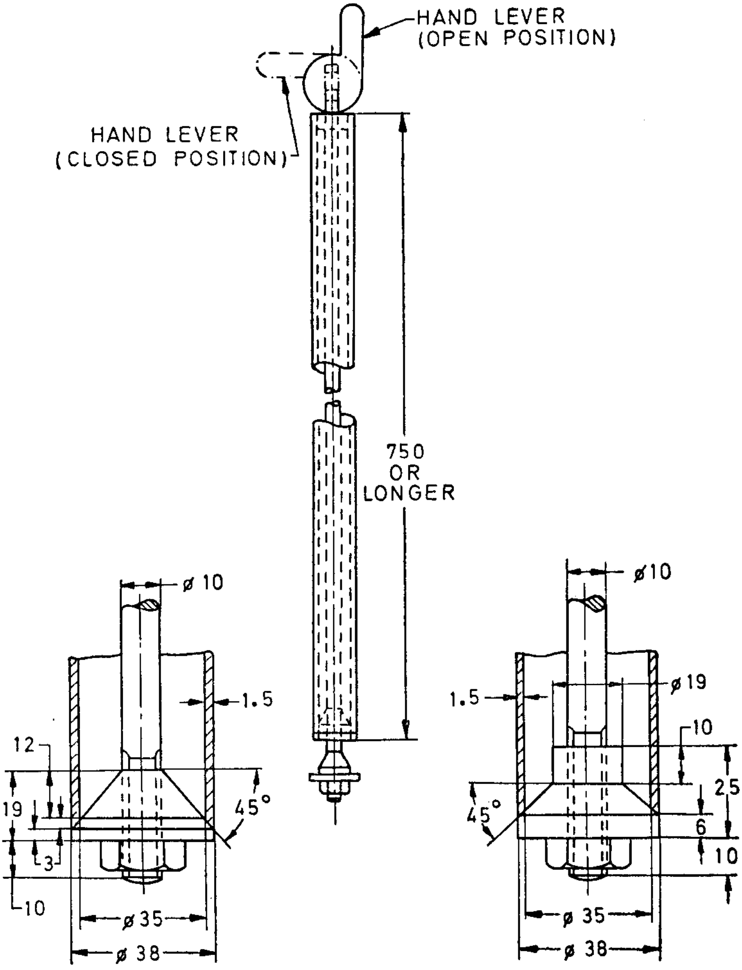
3.3.1.3 *Bottom-valved sampling tube*

a) *Design and construction* — Figure 3 shows the design of a bottom-valved sampling tube. Two types of bottom valves A and B are also shown in this figure. A length of 750 mm is suitable for most drums but a longer length may be required for other containers. The bottom valve should be checked to ensure a tight seal.

b) *Procedure for using bottom-valved sampling tube* — Lower the sampling tube, with bottom valve in closed position, into the container. When the tube reaches near the bottom of the container, open the valve by using the hand-lever and fill the tube with the liquid sample. Close the valve and withdraw the tube. Rinse the tube by draining the sample drawn. Once again collect the sample and transfer into the marked sample container.

3.3.1.4 *Thief* (*also known as sampling tube*)

a) *Design and construction* — Figure 4 gives some designs of thief. Suitable lengths of thief are 75 cm and 120 cm. Material of construction is usually glass. A glass rod with a hook on the upper side and stopper on the other side, is fitted into the thief. The stopper can either be of rubber or ground glass and it fits into the outlet of thief in the closed position, rendering the system leakproof. Outer diameter of thief can be 20 mm.

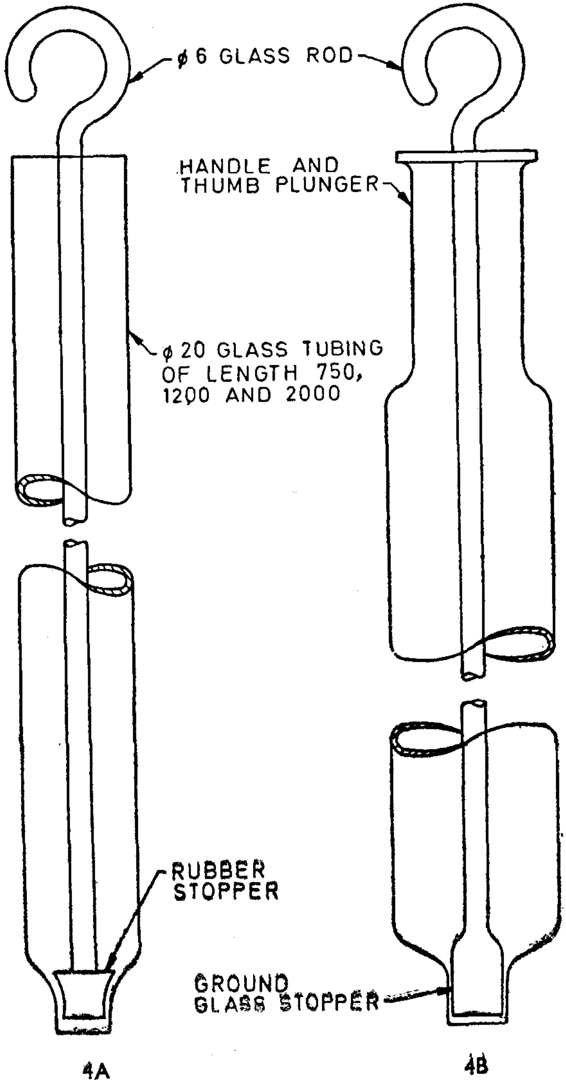


TYPE A

TYPE B

All dimensions in millimetres.

FIG. 3 BOTTOM-VALVED SAMPLING TUBE



All dimensions in millimetres.

Fig. 4 Sampling Thief for Mobile Liquids

b) *Method for using thief* — Lower the thief in the closed position into the container. When the thief nearly touches the bottom, pull the hook of the inner glass rod enabling the liquid to enter the thief from the bottom. After filling the thief, push the hook to close the thief and draw out the sampling equipment taking care to drain off the liquid adhering to outer portion of the thief. Transfer the sample thus collected into the marked sample container. In case of volatile liquids the sample container should be rinsed out with the liquid to be sampled so as to saturate the air in it with the vapour of the material.

3.3.2 *Sampling from Big Containers* — Big containers include storage tanks, tank trucks, shore tanks, ship tanks, barge tanks, tank-cars, vats, reaction vessels, etc. Tanks are sometimes quite deep, say more than 2 metres depth and in such cases the sampling equipment is suitably designed. The sampling equipment useful in such cases is thief dipper, dipper bottle, weighted can, sampling cage and bottom sampling apparatus. Actually, other sampling equipment described under **3.3.1** can also be used because in mobile and homogeneous liquids sample can be drawn from the surface of liquid.

3.3.2.1 *Thief dipper*

a) *Design and construction* — Thief dipper is so designed that the sample can be taken from within 13 mm of the bottom of the tank. A suitable dipper is illustrated in Fig. 5 and is constructed of metal tubes and castings, sufficiently heavy or weighted to sink into the liquid. The valve rod has a projecting stem and when this strikes the bottom of the tank, the top and the bottom valves open releasing air and admitting liquid. The thief should be suspended by wire or light chain and not by cord or any other fibrous material.

b) *Procedures for use of thief dipper* — Lower the dipper with the help of chain or wire in closed position slowly from the top of the tank. When the dipper touches the bottom of the tank, the top and bottom valves will open and liquid will flow in. When the container is full as indicated by discontinuation of air bubbles, pull the chain and draw out the dipper containing sample. Allow the superfluous liquid to drain off. Then transfer the samples collected into the sample container by lifting the rod thereby opening the bottom valve.

3.3.2.2 *Dipper bottle*

a) *Design and construction* — A typical design of a suitable dipper is shown in Fig. 6. The materials of construction and sizes for various components are given in the figure.

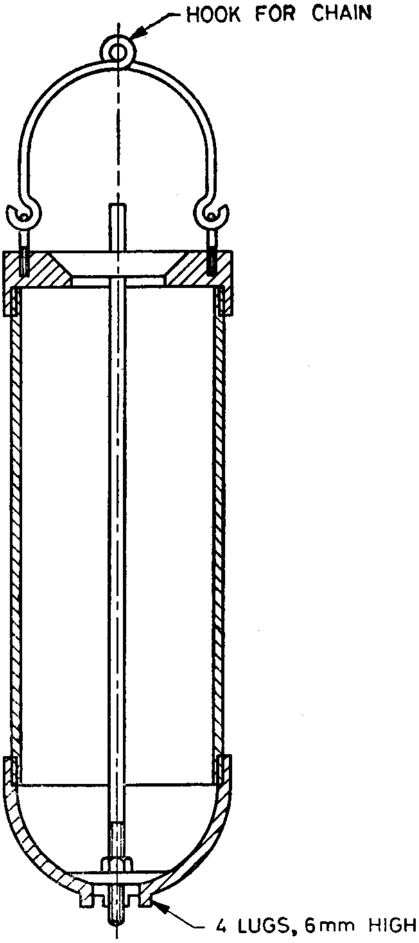
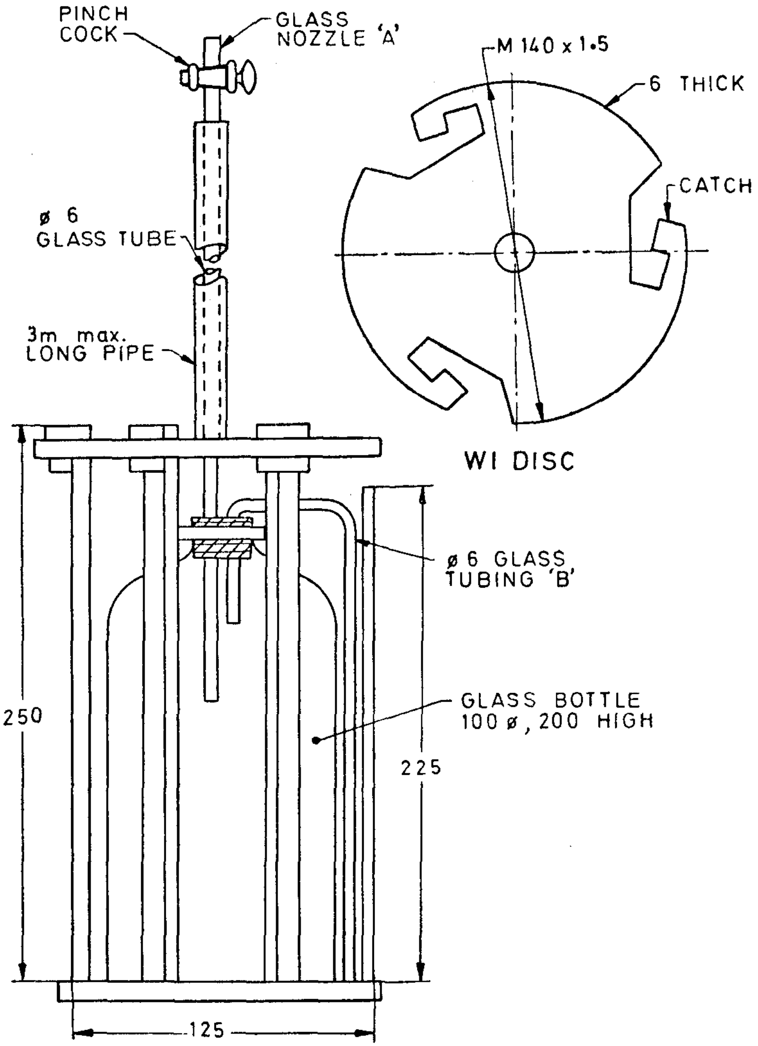


FIG. 5 THIEF DIPPER



All dimensions in millimetres.

FIG. 6 DIPPER BOTTLE

b) *Procedure for using dipper bottle* — Slowly lower the dipper bottle into the liquid with the outlet *A* open; a portion of the liquid from every point passes through the tube *B* into the bottle. Alternatively, if the sample is required at a particular spot (bottom, middle or top), the outlet *A* is kept closed until the desired depth is reached. Then open the outlet *A* and the sample is drawn entirely at that point. When the bottle is almost full or the required amount of sample fills the bottle, close the outlet *A* and pull out the bottle. Allow the superfluous liquid to drain off.

3.3.2.3 *Weighted can* (*or weighted bottle*)

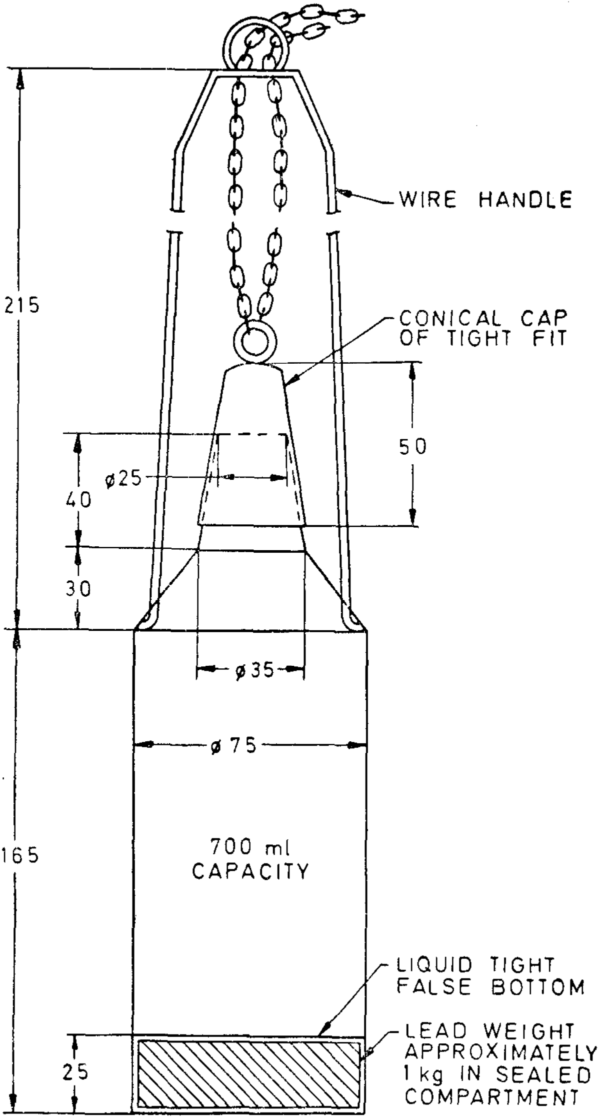
a) *Design and construction* — Figure 7 illustrates some designs of weighted can. It is normally of metal and weighted (say with lead) so that the closed empty can will sink easily into the liquid. This equipment should not be used for liquids which react with metals. The weighting material is fixed at the bottom of the can. A handle is provided for handling but alternatively a stand with a clamp to hold the can in position, can also be used. A chain or cord is attached with the stopper of the can.

b) *Procedure for using weighted can* — Close the can and lower it to the required depth (top, middle, lower) preferably as near the top surface of liquid as possible. Pull out the stopper with a sharp jerk of the cord or chain (non-sparking). When the air bubbles cease to rise indicating the can is full of sample, raise the apparatus at a uniform rate. The can should be nearly but not completely full of liquid sample. Immediately transfer, if necessary, the contents of weighted can into clean dry sample container of convenient size. In case it is difficult to see the rising bubbles, the can should be dipped for a period determined in a trial (for initial rinsing of apparatus).

3.3.2.4 *Sampling cage*

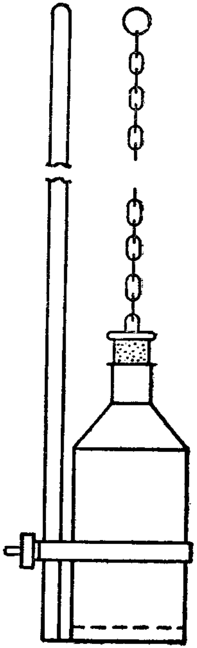
a) *Design and construction* — Some designs of the sampling cage are shown in Fig. 8. A metallic cage holds a sampling bottle in position. A chain or cord is attached to the stopper of the sampling bottle. The combined weight of the cage and bottle should be sufficient for them to sink into the liquid. The chain or cord should be of sufficient length to permit filling at any desired level. The sampling bottle can be introduced into the cage either from top (*see* Fig. 8A) or from the bottom (*see* Fig. 8B). In the latter case the bottle has a threaded neck which is screwed in a hole provided in the cage.

b) *Procedure for using sampling cage* — Fit the dry and clean sampling bottle into the cage and close the bottle. Lower the cage slowly into the container holding the sample, to the desired depth.


WIRE HANDLE
CONICAL CAP OF TIGHT FIT
215
50
ø25
40
30
ø35
ø 75
700 ml CAPACITY
165
LIQUID TIGHT FALSE BOTTOM
LEAD WEIGHT APPROXIMATELY 1 kg IN SEALED COMPARTMENT
25

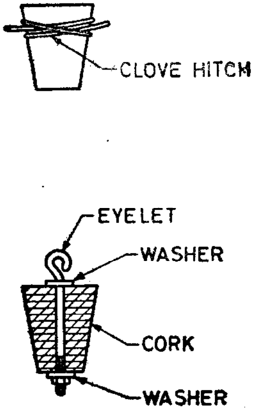
All dimensions in millimetres.

FIG. 7A WEIGHTED CAN

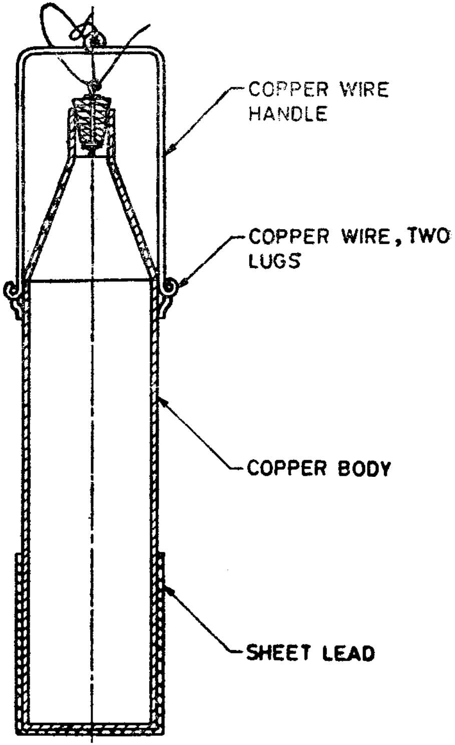


All dimensions in millimetres.

FIG. 7B SAMPLING BOTTLE OR CAN

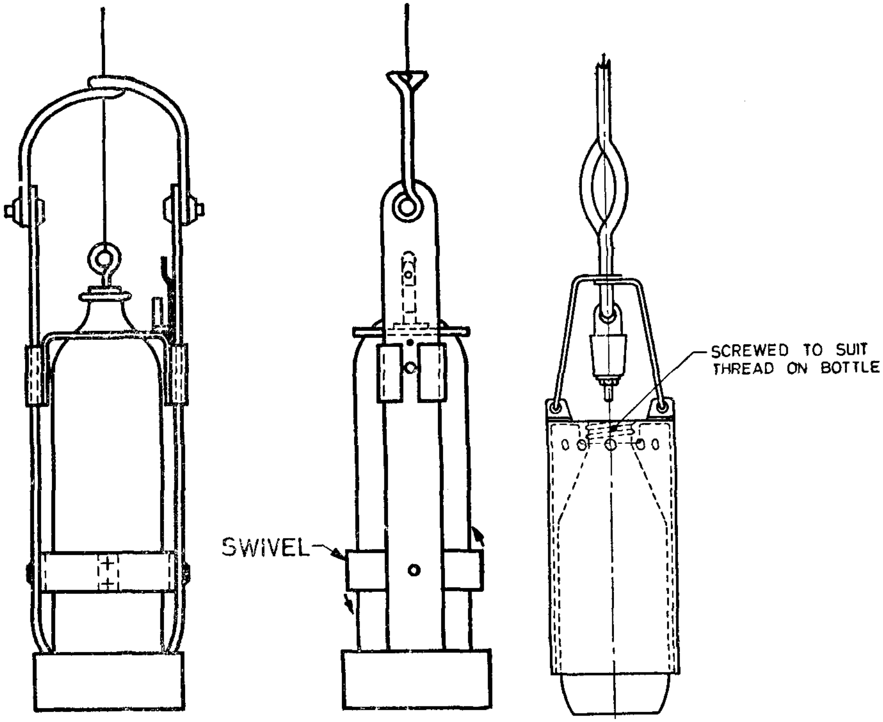


CORK ARRANGEMENTS



All dimensions in millimetres.

FIG. 7C ASSEMBLY FOR WEIGHTED BOTTLE OR CAN SAMPLING



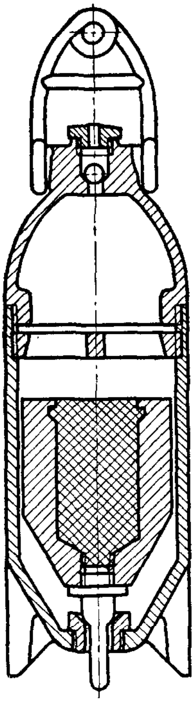
8A 8B

FIG. 8 SAMPLING CAGE

Open the stopper of the bottle by sharply jerking the chain. The liquid would start flowing into the bottle as evidenced by rising bubbles of air at the surface of the liquid. When these air bubbles cease to rise, lift the cage out at a uniform rate. Allow the liquid adhering to the cage and outer walls of the bottle to drain off. Remove the cage away from the tank and if necessary, transfer the contents of the bottle to a clean and dry marked sample container. Alternatively, the same bottle can be closed tightly (bottle almost but not completely full of sample) and marked for testing.

3.3.2.5 *Bottom sampling apparatus*

a) *Design and construction* — One type of bottom sampling apparatus is shown in Fig. 9. It consists of a cylindrical gun metal casing with a conical bottom fitted with four legs. Into the top of this casing is screwed a dome-shaped cover fitted with a ball-air release valve, with a handle to which the chain or cord of sufficient length can be attached. Inside the casing is inserted a square sliding weight, fitted at the lower end with a valve and projecting spindle which fits into the centrally positioned valve-seating in the bottom of the casing. A stop ring prevents the sliding weight from falling out when the casing is inverted with the top removed. A sampling apparatus of 70 mm diameter and 215 mm height can draw a sample of 160 ml liquid from a tank.



All dimensions approximate in millimetres.

FIG. 9 BOTTOM SAMPLING APPARATUS

b) *Procedure for using bottom sampling apparatus* — Lower the apparatus slowly until it just rests in an upright position on the bottom of the tank. When bubbles cease to rise indicating that the liquid sample has been collected, lift the apparatus out. Allow the adhering liquid to drain off and transfer the sample to a marked sample container. If it is difficult to see the rising bubbles, dip the apparatus for sufficient length of time based on experience.

3.4 Sampling Equipment for Materials which can be Rendered Mobile

3.4.1 Some materials can be rendered mobile by warming without deterioration. They include semi-solids (phenol), liquids solidified at low ambient temperatures and viscous liquids whose viscosity can be reduced significantly. In such cases the sampling apparatus should be as hot as necessary to prevent solidification in it. Glass parts of sampling equipment and sample containers should be heat resistant (for example, borosilicate). The sampling apparatus may be heated in a suitable hot-air oven at a temperature 5-10°C higher than that of the liquid to be sampled. Once the sample has been liquefied and maintained into this mobile state, any suitable sampling equipment described in **3.3** can be used.

3.4.2 The container holding the sample is heated until the contents are completely converted into a mobile liquid. While heating the container is provided with vents to prevent any build up of pressure during heating process. A drum may be heated in an open tank on a grid below which steam coil is fitted or alternatively by means of an electric heating blanket or jacket. The drum bung of stopper should be loosened to provide an effective vent before heating commences.

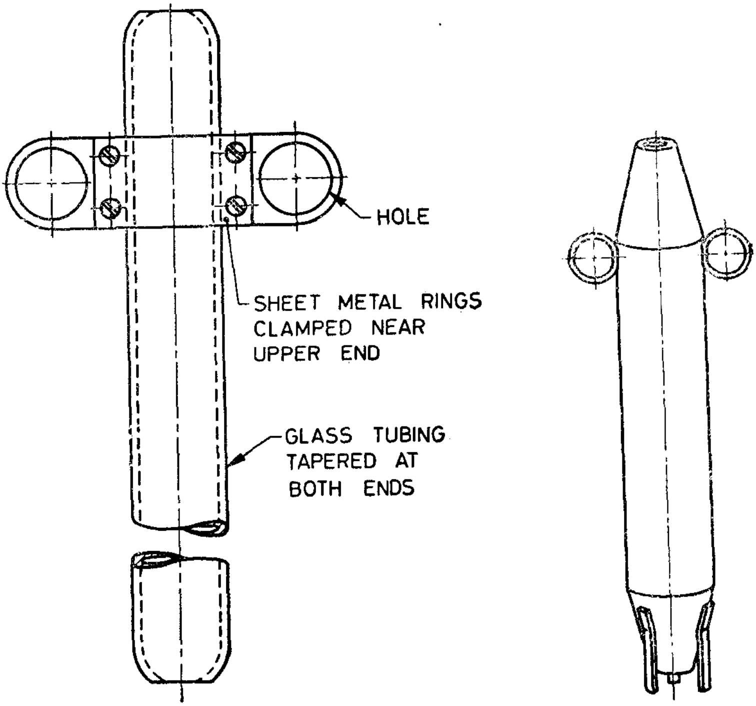
3.5 Sampling Equipment for Viscous Liquids

3.5.1 For liquids which cannot be rendered mobile (say by heating) but can flow out of their containers (lubricating oils, electrical insulating oil), sampling equipment like ladle (*see* **3.3.1.1**), bottom-valved sampling tube (*see* **3.3.1.3**), or weighted can (*see* **3.3.2.3**) can be conveniently used. Besides, specially designed sampling tube (thief) and pressure thief can also be employed.

3.5.2 *Sampling Tube (Thief) for Viscous Liquids*

3.5.2.1 *Design and construction* — Typical design of a glass sampling tube is shown in Fig. 10. The tube is made of thick glass and its capacity is minimum 500 ml. The lower end of the tube is ground slightly oblique and is long enough to reach the bottom of drum or barrel. A convenient internal diameter is about 25 mm. Two handles are fixed near the top of the tube which may be glass fused on or more conveniently made of sheet metal clamped on. The use of a metal thief is also permissible (*see* Fig. 10B). One form consists of a sheet metal tube fitted at each end with an open cone-shaped cap and round the lower end three legs equally spaced are fitted to hold the opening 3 mm from the bottom of the container.

3.5.2.2 *Procedure for using sampling tube* — The details of procedure are the same as described under **3.3.1.2**(b). It takes more time to fill the viscous sample into the tube. After the tube full of sample is drawn


HOLE
SHEET METAL RINGS CLAMPED NEAR UPPER END
GLASS TUBING TAPERED AT BOTH ENDS

10A Glass Thief

10B Metal Thief

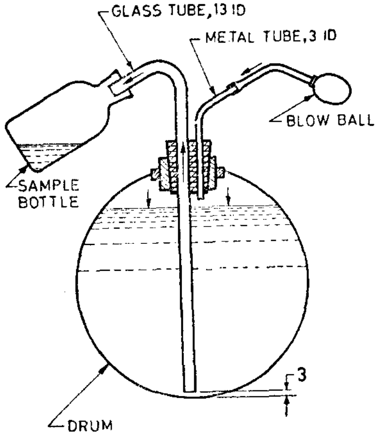
FIG. 10 SAMPLING TUBE FOR VISCOUS LIQUIDS

out, sufficient time should be allowed for the liquid on the outside of the tube to drain off or alternatively the outside of the apparatus may be wiped.

3.5.3 *Pressure Thief*

3.5.3.1 *Design and construction* — A glass tube having an internal diameter of 13 mm with one end extending to within3 mm of the bottom of the container and the other end bent at a suitable angle, is fitted into a rubber stopper. Another tube shorter in length and 5 mm internal diameter is also bent and fitted into the same stopper. A blow ball is attached to the small tube. The assembly of apparatus is shown in Fig. 11.

3.5.3.2 *Procedure for using pressure thief* — Clean and dry the glass parts and fit the assembly as shown in Fig. 11. The longer tube is inserted into the sample bottle. Press the blow ball so that the liquid rises through the



All dimensions in millimetres.

FIG. 11 PRESSURE THIEF

longer tube and fills into the sample bottle. When the sample bottle is almost full, remove the stopper carefully. The liquid filled in the longer tube slowly falls back into the container. Clean the tubes with suitable solvents and dry. The sample bottle is immediately stoppered tightly and marked for testing.

3.6 Sampling Equipment for Multi-Phase Liquids

3.6.1 Sometimes a liquid sample has more than one phase, liquid or solid. The separate phase may be suspended or may settle quickly. The second phase may form due to decomposition of the sample or it may be a contaminant. The sampling equipment suitable for multi-phase liquid systems includes open-sampling tube (*see* Fig. 2), bottom-valved sampling tube (*see* Fig. 3), etc. In case of liquid-solid heterogeneous systems thorough mixing is essential so that the solid particles get uniformly distributed in the system. Immediately after homogenisation of the system closed-end sampling tube [described in Fig. 3 of IS : 8883 (Part II/Section 1)-1978*], ladle (*see* Fig. 1), weighted can (*see* Fig. 7) or sampling cage (*see* Fig. 8) is introduced into the container. Time lapse between mixing and sample collection should be the minimum possible. The sampling can or cage if used for this purpose, should have wider mouth than normal.

3.6.2 *Procedure for Using Sampling Tubes for Liquid-Liquid Multiphase Systems* — Open-end sampling tube (*see* Fig. 2A and 2B) is lowered into the container very slowly to cause minimum disturbance in the liquid-liquid multiphase system. The upper end of the tube is not closed with thumb but kept open. As far as possible at any stage of insertion of sampling tube into the container, the level inside the tube should be the same as that at the outside surface of the liquid. When the tube reaches the bottom of the container, the upper end is closed with thumb and the tube withdrawn. The sample collected is transferred to the marked sample container.

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*Methods of sampling of chemicals and chemical products: Part II Sampling equipment, Section 1 For solids.

3.7 Sampling Equipment for Liquefied Gases — In the case of gases which are stored or handled as liquids, the composition of the gaseous phase in contact with the liquid may differ from that of the liquid phase because more volatile material is largely present in the gaseous phase. For sampling of gaseous phase the procedures shall be as laid down in IS : 8883 (Part II/Sec 3)-1979*. Any suspended matter present in liquefied gases cannot be detained by sampling of gaseous phase. The sampling of liquefied gases is sometimes a potential hazard. The areas where samples are being taken should be adequately ventilated; preferably the sampling may be done in the open.

3.7.1 *Sampling from a Small Container* — Sample bomb of various capacities, for example, 1 litre, 4 litres can be employed. Alternatively, if the pressure is small, the liquid is allowed to be driven into a Dewar vessel.

3.7.1.1 *Sample bomb up to one litre capacity*

a) *Design and construction* — Typical design of stainless steel gas sampling bomb is shown in Fig. 12. The bomb is made of stainless steel (04Cr17Ni12Mo2) and designed to withsand sufficient pressure. The bomb is in the form of a long cylinder with steel needle valves at each end.

Suitable dimensions of the bomb are as under:

Capacity	*Approximate Dimensions of Body*	
(ml)	Diameter (mm)	Length (mm)
100	25	200
200	38	200
500	41	400
1 000	57	400

Stainless steel needle valves may be of 6 mm size and tested to twice the working pressure to which it may be subjected.

b) *Procedure for using gas sampling bomb* (*1 litre capacity*) — Vent any liquid from the bomb and connect the bomb in an upright

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*Methods of sampling of chemicals and chemical products: Part II Sampling equipment, Section 3 For gases (*under preparation*).

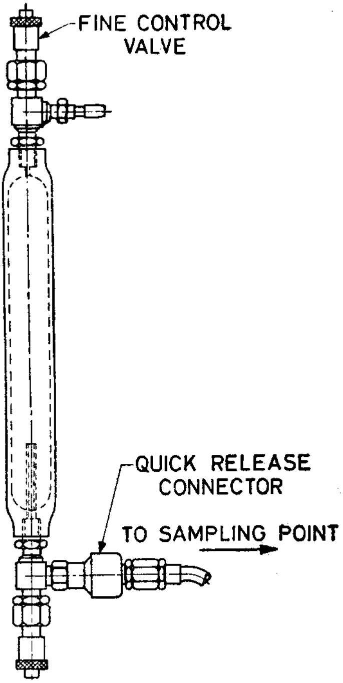


FIG. 12 GAS SAMPLING BOMB, 1 litre CAPACITY

position to the source at a point at least 100 mm below the liquid level so that liquid may enter the bottom of the bomb. Open the outlet and inlet valves. If possible, cool the sample bomb and connector to below the boiling point of the sample with a suitable cooling medium. Open the sample source valve (say from cylinder) and if cooling is impracticable, maintain the pressure in the bomb high enough to prevent the liquid boiling, by partially closing the outlet valve. When the liquid starts flowing through the outlet valve, quickly close this valve, then close the inlet valve and finally close the sample source valve. Immediately invert the bomb, open the outlet valve and expel the liquid filled. This operation removes contamination from a previous sample, cools the apparatus and ensures representative sampling. Close the outlet valve, reinvert the bomb, support it in an upright position and commence taking the sample by opening the inlet valve momentarily from time to time until liquid appears. Close the outlet, inlet and sample source valves quickly and in that order. Disconnect the bomb from the sample source. To provide safety ullage, immediately support the bomb in an upright position, open the bottom valve and vent approximately 20 percent of the contents. Close the valve and confirm by weighing that the correct ullage has been obtained.

3.7.1.2 *Gas sampling bomb of 1 litre capacity with ullage chamber*

a) *Design and construction* — Fig. 13 shows the design of gas sampling bomb of 1 litre capacity with ullage chamber. The bomb has two compartments in the volume ratio of 1 : 4 connected by means of a valve. The material of construction is stainless steel and system (also the valves) tested for withstanding twice the working pressure. With this type of gas sampling bomb, it is possible to provide 20 percent ullage precisely.

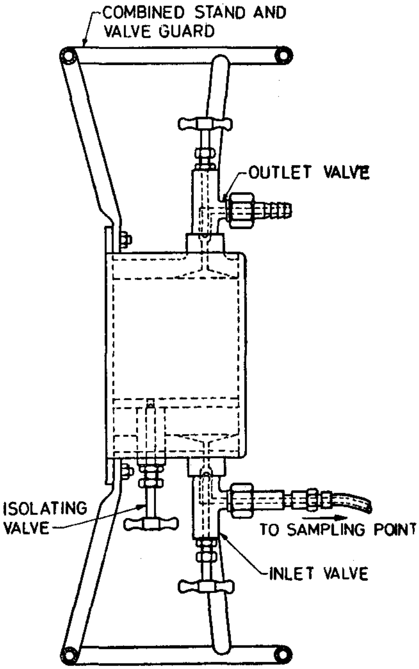


FIG. 13 GAS SAMPLING BOMB, 1 litre CAPACITY, WITH ULLAGE CHAMBER

b) *Procedure for using gas sampling bomb with ullage chamber* — Vent any liquid present in the bomb. Connect the bottom valve to the sample source so that the liquid may enter the bomb. With the outlet interconnecting and inlet valves open, fill the bomb completely with liquid by opening the sample source valve and manipulating the outlet valve to prevent the liquid from boiling. When the liquid appears at the outlet valve quickly close it, then close the inlet and sample source valves. Disconnect the bomb from the sample source valves. Invert the bomb, open the bottom valve and expel the liquid from the bomb. Reinvert the bomb and reconnect it to the sample source, open the sample source valve and refill the bomb as before but with the outlet valve closed, opening it intermittently until liquid appears. As soon as the liquid appears, close the outlet, inlet and then the sample source valve and disconnect bomb quickly. Immediately close the interconnecting valve, open the inlet valve and empty the liquid from the smaller compartment.

Close the valve when liquid ceases to emerge and then quickly reopen the interconnecting valve to provide the necessary ullage.

3.7.1.3 *Gas sampling bomb of 2 ml capacity*

a) *Design and construction* — Typical design of 2 ml capacity sampling bomb is shown in Fig. 14. There is a nylon tube connected to two identical valve-cum-inlet stainless steel assemblies. This bomb is particularly useful where small amount of sample is available.

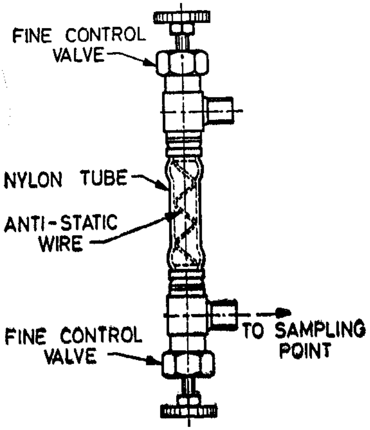


FIG. 14 GAS SAMPLING BOMB, 2 ml CAPACITY

b) *Procedure for using 2 ml capacity gas sampling bomb* — Open inlet and outlet valves of the gas sampling bomb and connect it in an upright position with the inlet valve at the bottom to the liquid sample source. Purge until liquid appears at the outlet. Close the outlet valve and after about 10 minutes (to establish equilibrium) close the inlet valve. Finally close the sample source valve and disconnect sample bomb. Support the bomb in an upright position, open the bottom valve and vent approximately 20 percent of contents of bomb. Close the valve and transfer to the laboratory for testing. In the laboratory the sample may be completely vaporized in a vessel of such size as to contain the sample at nearly atmospheric pressure.

3.7.1.4 *Gas sampling bomb of 0·5 ml capacity*

a) *Design and construction* — Fig. 15 shows the design of 0·5 ml capacity sampling bomb along with an attachment of vaporizing equipment. The bomb consists of two small size fine control steel valves connected together with the help of a smaller bore tube for sample collection.

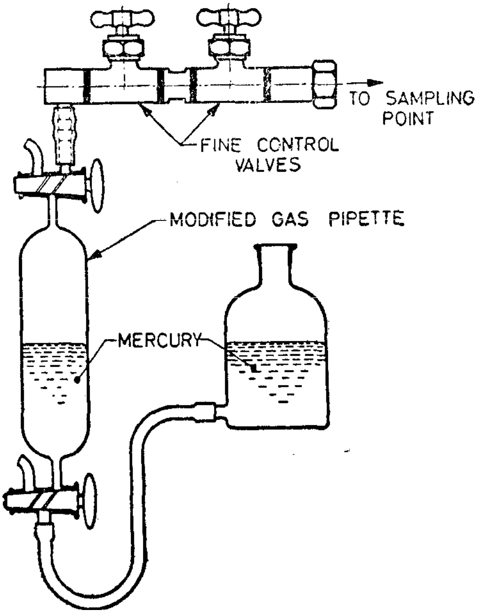


FIG. 15 GAS SAMPLING BOMB, 0·5 ml CAPACITY

b) *Procedure for using 0·5 ml capacity gas sampling bomb* — Place the sampling bomb in an upright position. Connect the bomb with the inlet valve at the bottom to the liquid sample source. Purge until liquid appears at the outlet valve. Close the outlet valve and after about 10 minutes (to establish equilibrium) close the inlet valve. Close the sample source valve and disconnect sampler. Connect to the sample bomb a mercury filled gas pipette (vaporizing equipment) attached by flexible tubing to a mercury reservoir. Remove air in the line by manipulating reservoir and stopcock. With both stopcocks of the pipette in the appropriate position, lower the mercury reservoir and open the outlet valve of the sample bomb to vaporize the liquid sample collected in the gas pipette. Close both stopcocks of the pipette and disconnect it from mercury reservoir. The capacity of the gas pipette should be large enough to contain all the vaporized sample at approximately atmospheric pressure.

3.7.1.5 *Metal Dewar apparatus*

a) *Design and construction* — A typical metal Dewar apparatus is shown in Fig. 16. It consists of a narrow necked metal Dewar vessel fitted with a screw cap carrying separate valves for filling, venting, and the withdrawal of sample through a copper capillary tube. This apparatus is used where sufficient sample is available as some losses occur during sampling. Loss of more volatile component takes place, leaving behind liquid enriched in less volatile components. Thus the equipment has some limitations and disadvantages for collecting liquid samples from liquefied gases. On the other hand it is safer to use it for gases liquefied at low temperatures.

b) *Procedure for using metal Dewar apparatus* — Ensure that the apparatus is clean and dry. The sample is obtained either by direct filling or cap filling. Direct filling method is preferred where contact of the sample with atmosphere can be tolerated, because with a high rate of filling there is less loss of volatile constituents by evaporation. Remove the cap from the apparatus. Place a 10-mm dip tube in the open neck and connect it to the sampling point valve. Open the sampling valve fully and close it as soon as sufficient liquid has been collected. Remove the dip tube, open the vent valve of the cap and screw the cap into the apparatus. The cap-filling method should be used where contact with the atmosphere has to be kept to the minimum, but since the rate of filling will be slow, losses of more volatile constituents by evaporation will be significantly high. According to this method the cap is screwed on to the neck of the apparatus and all the valves are closed. Connect the filling valve to the sample source valve. Open the vent valve and filling valve on the cap and the sample source valve, in that order. The liquid is filled at a slow rate and when the required volume of the liquid has been filled, close the sample source valve and then the filling valve. Disconnect Dewar apparatus from the sample source valve. The outlet from the

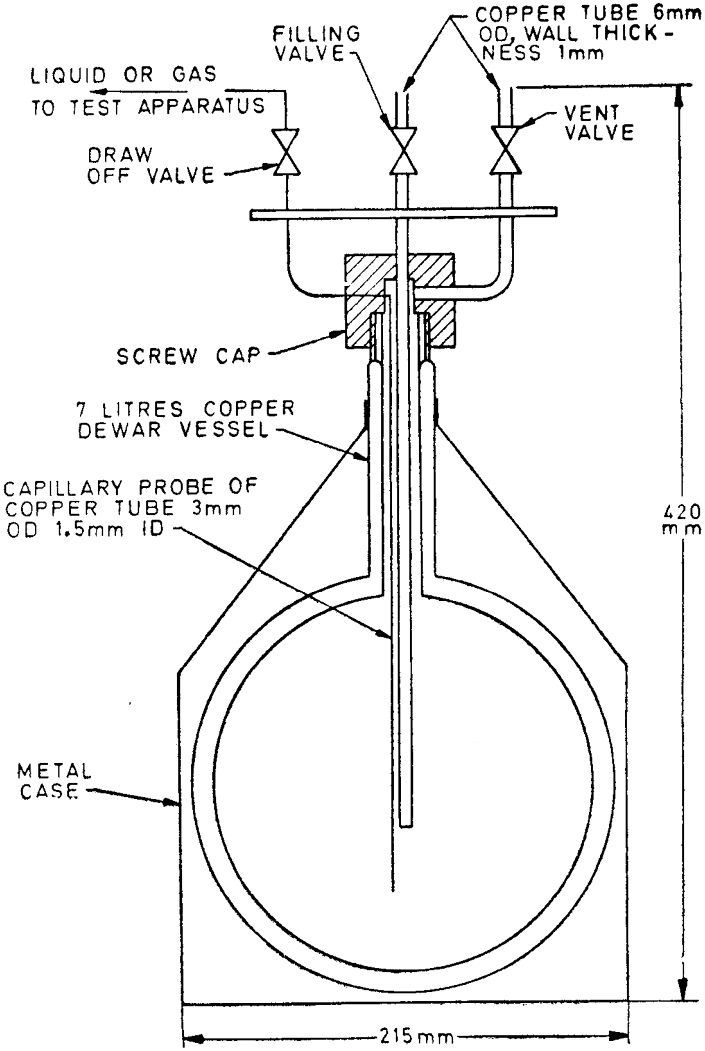

COPPER TUBE 6mm OD, WALL THICKNESS 1mm
FILLING VALVE
LIQUID OR GAS TO TEST APPARATUS
VENT VALVE
DRAW OFF VALVE
SCREW CAP
7 LITRES COPPER DEWAR VESSEL
CAPILLARY PROBE OF COPPER TUBE 3mm OD 1.5mm ID
420 mm
METAL CASE
215 mm

FIG. 16 METAL DEWAR APPARATUS

vent valve should be checked occasionally during filling to ensure that it has not been blocked by solidified material. To prevent dangerous build-up of pressure in the Dewar apparatus it should always be vented except at the time of drawing a portion of sample for testing as this will be rich in the more volatile components. Gas issuing from the vent valve should not be used for testing. To obtain a representative test portion, it is necessary to force liquid out of apparatus through the capillary probe and then ensure that this is completely converted into gas. For this purpose connect an empty gas cylinder to the filling valve via a suitable two stage reducing valve and a pressure gauge. Connect a metal capillary tube, partly twisted into a coil, to the cylinder sampling valve. The coil is immersed in a vessel of hot water. The open end of this tube is connected to the required test apparatus. Close the vent valve, open the filling valve and sampling valve of the cylinder. Apply sufficient pressure from the gas cylinder to produce a satisfactory rate of flow of gas from the capillary tube. Renew the hot water round the coil if necessary. The pressure applied should not normally exceed **70** kN/m^2.

3.7.2 *Sampling from a Large Container* — Gas sample bombs as described under **3.7.1** can be used. These bombs are up to 1 litre capacity and when larger amount of sample is required special gas sample bombs of higher capacity (say 4 litres or 25 litres) can be used. Metal Dewar apparatus can also be used (*see* **3.7.1.5**).

3.7.2.1 *Gas sampling bomb of 4 litre capacity*

a) *Design and construction* — The design of 4 litre capacity gas sampling bomb is shown in Fig. 17. There is a provision for ullage of 20 percent. A pressure gauge is also attached with the bomb to indicate the pressure developed in the bomb.

b) *Procedure for using 4 litre gas sampling bomb* — Vent any liquid from the bomb. Support the bomb in an upright position and connect the inlet valve to the source valve. Open the outlet and inlet valves. If possible cool the bomb and connector to a temperature below the boiling point of the sample with a cooling medium. If such cooling is impracticable, maintain the pressure in the bomb high enough to prevent the liquid from boiling by partially closing the outlet valve. When liquid appears at the outlet valve, quickly close this valve, then the inlet valve and finally the sample source valve. Immediately invert the bomb, open the outlet valve and expel the liquid content. Close the outlet valve and reinvert the bomb. Again open the inlet valve and then the sample source valve. Open the inlet valve momentarily from time to time until liquid appears. Close the outlet valve, then inlet valve and finally the sample source valve quickly. Disconnect the bomb from the sample source and provide safety ullage (say 20 percent) immediately by opening the inlet valve and closing it again.

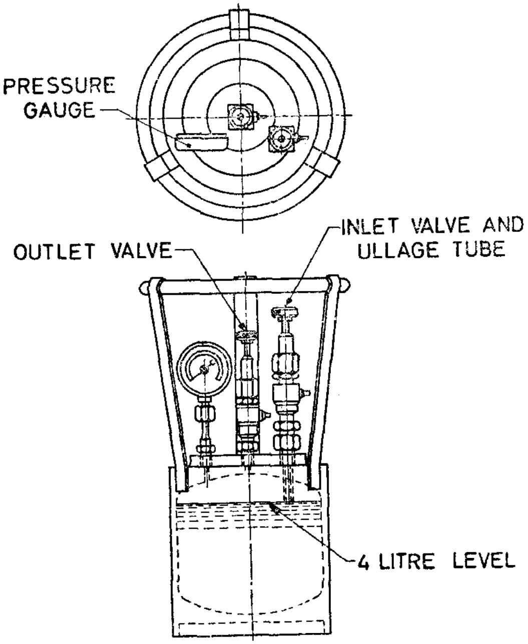


Fig. 17 Gas Sampling Bomb, 4 litre Capacity

3.8 Sampling Equipment for Liquids Requiring Special Handling (Hazardous Liquids) — Liquids requiring special handling can be classified as under:

a) Liquids that react with one or more components of the atmosphere;

b) Reactive or unstable liquids; and

c) Flammable liquids.

3.8.1 *Sampling Equipment for Liquids Reactive to Atmosphere* — The liquid sample can be reactive towards oxygen, water, carbon dioxide, light, temperature, dust and biological bodies. Near industrial plants, atmospheric pollutants can also be reactive towards the sample. To prevent the action of oxygen, moisture and carbon dioxide sampling may be done in a dry and inert atmosphere or it may be covered by a lower density non-reactive liquid. Nitrogen is an example of inert gas. Thus a sampling equipment which can be purged by a gas is required. In one method the small tubing may be inserted through the inlet of the container holding the sample and dipped into the liquid. The other end of the tube is connected to a source of inert gas. The gas is bubbled through the liquid which provides an inert atmosphere over the surface of the liquid. Then an open sampling tube (*see* **3.3.1.2**) purged with the same inert gas can be carefully used to draw the sample. Preferably, a weighted can or cage (*see* **3.3.2.3** and **3.3.2.4**) thoroughly purged with inert gas can be employed for sampling. After drawing the sample, the bottle or can is immediately stoppered and marked for testing. The sample should not be transferred from the weighted can or bottle (in the cage) to any sample container. To avoid hygroscopic action of atmospheric humidity a dry gas (say nitrogen) is passed through the sample, the gas being dried by a trap of silica gel or calcium chloride. Equipment for dry sampling should be dried before use. Weighted can or sampling cage can be used but with proper care. Alternatively pressure thief (*see* **3.5.3**) or vacuum sampling methods can be used to avoid interference of atmospheric oxygen, carbon dioxide and moisture.

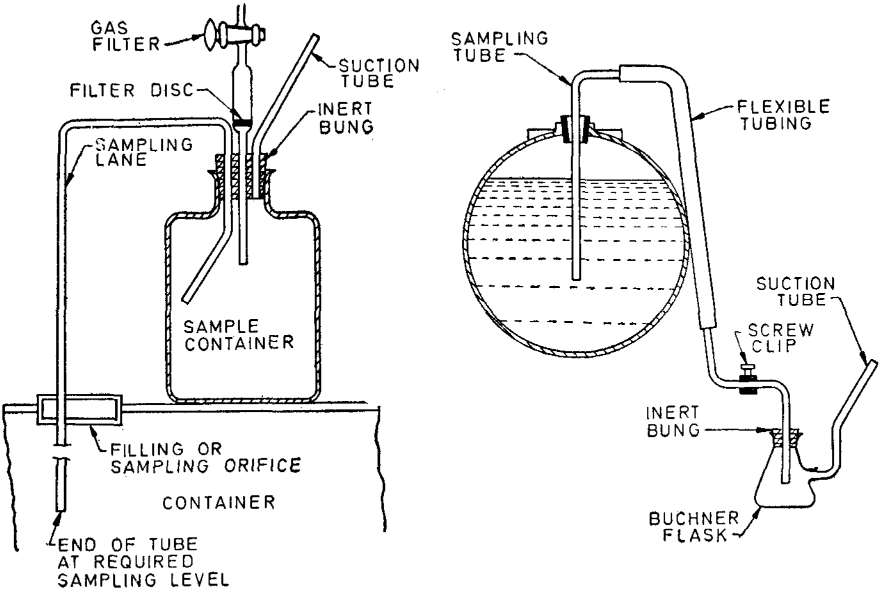
3.8.1.1 *Vacuum sampling apparatus*

a) *Design and construction* — Two typical types of apparatus (*A* and *B*) are shown in Fig. 18. The suction tube should incorporate a trap to protect the pumping system in the event of the sample container becoming full. Type *A* is used whenever the sample collecting bottle cannot be placed at a level lower than the surface of the liquid to be sampled. Type *B* avoids the lifting of all the equipment to the top of the containers such as tankers.

b) *Method for using vacuum sampling apparatus*

   1) *Method for Type A* — Clean and dry the sampling apparatus. Insert the stopper having an inlet for sample, an outlet for suction and a gas filter, into the neck of the sample container. Connect the outlet tube to the suction pump and close the gas filter tap. Evacuate the sample container until the liquid begins to flow in the sample container through the inlet tube the other end of which is dipping into the liquid. When sufficient sample has been collected open the gas filter tap. The level of the liquid in the sample container should be below the end of the inlet tube, otherwise the sample will siphon back when suction is stopped.

   2) *Method for Type B* — Clean and dry the equipment which in this case is a Buchner flask. The sampling tube is inserted into the flask through a stopper and the other end of the tube is dipping into the container holding the sample. Open the screw clip and connect the suction tube to the suction pump. Evacuate the flask until the liquid begins to flow. When sufficient



18A Type A

18B Type B

Fig. 18 Typical Vacuum Sampling Apparatus

liquid collects in the flask the screw clip is closed. Alternatively, the system can be used as a siphon after the initial vacuum fills the sampling tube. Close the screw clip for a while and connect the suction tube to a sample container. Open the screw clip and start the siphon till the required amount of sample is collected in the sample container. The flow of the liquid can be controlled by the screw clip. Finally break the siphon by lifting the sampling tube out of the liquid in the container.

3.8.1.2 *Sampling equipment when a liquid is used as a cover over reactive liquids* — If covered by a liquid the sampling should be carried out beneath the surface of the covering liquid. Even the sample container should also contain the covering liquid. It has to be ensured that the covering liquid is not soluble in the sample. A bottom-valved sampling tube (*see* **3.3.1.3**) can be used for drawing the liquid sample beneath the covering medium, and transferred to the sample container, already containing the covering liquid. The sample tube is made to release the sample below the surface of the covering liquid in order to avoid any atmospheric contact of the sample. Alternatively, the sample container filled with the covering liquid can be used in a sampling cage (*see* **3.3.2.4**).

3.8.1.3 *Sampling equipment for liquids when dust, industrial gases and bacteria are present in the atmosphere* — Dust and gaseous atmospheric pollutants can be avoided by supplying clean air at the sampling point, and if possible enclosing the sampling area temporarily. Alternatively, the conditions described under **3.8.1.1** and **3.8.1.2** can be followed. To prevent bacterial and fungal contamination it is necessary to conduct sampling under aseptic conditions including sampling apparatus, sample container and the environment. Usual sampling equipment depending upon the type of liquid to be sampled can be used.

3.8.1.4 *Sampling equipment for liquids sensitive to light* — Liquids affected by light should be sampled in minimum illumination. The sampling equipment required is same as described in the previous clauses, depending upon the type of liquid. The sample container in such cases may be amber-coloured glass bottle or one with black paper or paint around it.

3.8.1.5 *Sampling equipment for liquids sensitive to change in temperature* — Liquids which change in their composition with change in temperature should be sampled at a temperature at which they are normarlly stored. Sampling equipment described under **3.3** can be used. After collection of sample, the sample container should be stored at temperature less than the limit at which liquid sample decomposes.

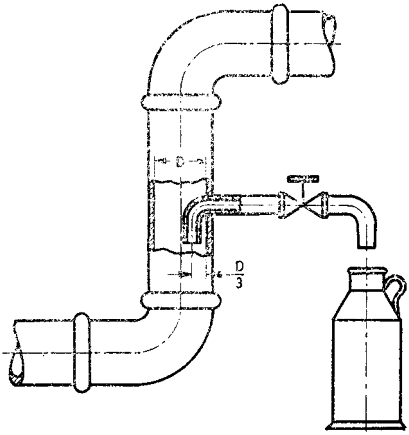
3.8.2 *Sampling Equipment for Unstable Liquids* — A liquid may be unstable due to reactive nature of its own components or conditions of environment. The latter part has been discussed under **3.8.1**. The liquid may also be reactive towards the material of sampling equipment. In any case the nature of liquid sample to be drawn should be clearly known and sampling precautions taken accordingly. For instance, liquids which decompose with time should be sampled and tested before such time. Material of construction of the sampling should also be selected considering the reactivity of the liquid sample. Warning should also be displayed on the sample container if the sample is unstable or hazardous. Bottom-valved sampling tube (*see* **3.3.1.3**) or preferably vacuum sampling apparatus (*see* **3.8.1.1**) can be used for sampling of such unstable and reactive liquids.

3.8.3 *Sampling Equipment for Flammable Liquids* — The vapour pressure of some liquids is such that there will be an explosive mixture when air is present. All sampling apparatus used should be made of material not conducive to formation of a spark. The discharge of static electricity can also give rise to sparks which, if they possess sufficient energy, can ignite the vapour of a flammable liquid. Bottom-valved sampling tube (*see* **3.3.1.3**) or vacuum sampling apparatus (*see* **3.8.1.1**) can be used under strict precautions.

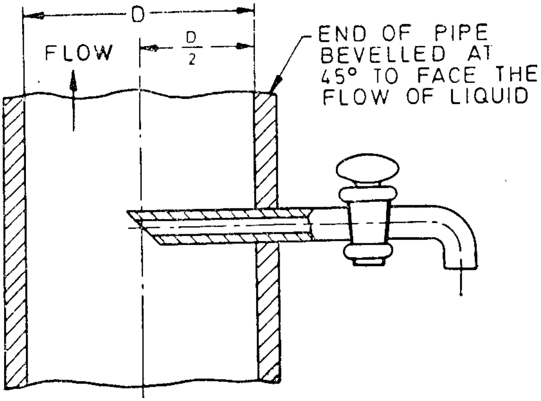
3.9 Sampling Equipment for Liquids in Motion — When the liquid is flowing through a pipeline, the sample can be collected from the open end of pipe. The alternative sampling equipment is pipeline probe which may be a permanent attachment to the pipeline, filling lines and transfer lines.

3.9.1 *Pipeline Probe*

3.9.1.1 *Design and construction* — Typical designs of some pipeline probes are shown in Fig. 19. In one case (*see* Fig. 19A) a pipe of approximately 13 mm internal diameter is fitted to extend internally one-third of the distance across the diameter of pipeline. The inner end of the pipe is bent at an angle of 90° to face the stream with the external end provided with a suitable cock. Whenever possible, the pipe should be installed in a



19A Type A



TYPE B

19B

FIG. 19 PIPELINE PROBE

rising section of the line and on the delivery side of the pump, but if there is no rising section, the pipe should enter the line above the horizontal diameter. To keep the orifice free from dirt, screens of area larger than the openings may be placed around the pipes. A pipe cap with a suitable orifice may be substituted for the cocks. In the other design (*see* Fig. 19B), a 12-mm bore pipe is fitted preferably on the discharge side of any pump. The pipe extends into the centre of the pipeline and its inner end bevelled at 45° to face the flow of liquid which should be upwards in the pipeline. The length of the pipe outside the pipeline should be as short as possible and provided with a cock. The material of construction of the pipe and cock may be stainless steel, plastics or any other material not reactive to the liquid sample.

3.9.1.2 *Procedure for using pipeline probe* — Turn the plug cock so that a steady stream is drawn from the cock at such a rate that about 0·1 percent of the product or 180 litres, whichever is less, is diverted through the line during the drawing of sample. When sampling semi-liquids, heat the sample contained in the receiving coils to a temperature just sufficient to keep the material in liquid state. The sample container should be immediately stoppered and marked for testing.

3.9.2 *Three-Way Stopcock* — If the pipeline has no open end to allow the sample collection and the pipeline diameter is small for the probe to be fitted, a three-way cock should be fitted into the pipeline so that the whole of the flow can be diverted for a set period into the sample container. A funnel can be placed in a sample container and held under the exit stream for a short period ensuring that the whole of the stream is caught by the funnel.